Methods for Preparing and Characterizing Granular Materials for Electron Yield Measurements

Tom Keaton
Utah State University

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METHODS FOR PREPARING AND CHARACTERIZING GRANULAR MATERIALS FOR
ELECTRON YIELD MEASUREMENTS

by

Tom Keaton

A report submitted in partial fulfillment
of the requirements for the degree

of

Master of Science

in

Physics

Approved:

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JR Dennison, Ph.D.
Major Professor

______________________
Jim Wheeler, Ph.D.
Committee Member

______________________
Jan Sojka, Ph.D.
Committee Member

UTAH STATE UNIVERSITY
Logan, Utah

2023
ABSTRACT

METHODS FOR PREPARING AND CHARACTERIZING GRANULAR MATERIALS FOR ELECTRON YIELD MEASUREMENTS

by

Tom Keaton, Master of Science

Utah State University, 2023

Major Professor: JR Dennison
Department: Physics

This work presents a systematic study on sample preparation methods and accuracy of electron yield (EY) measurements of highly insulating, granular materials. EY measurements of highly insulating materials, especially those with high EY, are challenging due to the effects of sample charging even for very low fluence electron probe beams. EY measurements of particulates are complicated by: (i) roughness effects from particulate size, shape, coverage, and compactness; (ii) particle adhesion; (iii) substrate contributions; and (iv) electrostatic repulsion and potential barriers from charged particles and substrates. Numerous methods were explored to rigidly affix particles on conducting substrates at varying coverages for accurate EY measurements. Gravimetric deposition of particles suspended in deionized water onto standard scanning electron microscopy (SEM), aluminum backed, graphitic carbon tape with a carbon infused, acrylic-based, conductive adhesive top layer, proved the most successful method, with robust results for ranges of particle sizes, shapes, and coverages. To mitigate potential electrostatic lofting effects of charged particulates, less adhered particles were removed with dry nitrogen jets and applied high electric fields prior to EY measurements. Particle sizes were determined via laser diffractometry, while SEM measurements were used to determine fractional coverage of adhered particles. Low fluence, pulsed electron probes (3-5 μs at 1-30 nA-mm^2) used 10^0 to 10^2 electrons
per pulse per particle to measure EY with minimal charging effects. Surface charge accumulation from each pulse was dissipated between pulses with 1-2 s bursts of ~4.9 eV photons from a UV LED and electrons from a flood gun; 3 to 6-hour thermal annealing of the samples at 310 to 340 K could also be used intermittently to dissipate deeper dielectric charging. Preliminary studies of highly insulating, 67±23 μm sized, angular Al₂O₃ polishing compound particles adhered to graphitic carbon conductive tape from 0% to ~100% coverage are presented to demonstrate the effectiveness of these methods. Results of high accuracy EY tests using these methods have important applications in lunar dust and asteroid technologies and lofting, electrostatic dust agglomeration in space, granular and aerosol coatings for spacecraft charge mitigation, and many coating, contamination and roughening issues applied to a wide variety of fields subject to charging.

(56 pages)
This research investigates the methodology behind creating and characterizing homogeneous, insulative granular compounds, for electron yield (EY) measurements. For our specific application, we utilized a major Lunar dust component: Aluminum oxide. EY induced charging of dusty surfaces are of direct importance to space-based goals more specifically relevant to NASA objectives because dust is the primary cause of accelerated equipment degradation and has adversely affected astronauts’ health. If we wish to return to the moon to stay, or explore other airless, dusty bodies in our solar system for prolonged periods of time, we must first understand the electrodynamical properties of their regolith materials. Although such issues have been recognized since the Apollo era, previous experimental work has failed to acquire information relevant to Lunar dust’s fundamental charging properties. Left unaddressed, government and commercial entity efforts to return to the Moon will continue to face hazards due to electrostatic charging of dust. Therefore, this work addresses the many technical challenges faced prior to a measurement being conducted in the first place, as these parameters must both be accounted for or minimized to produce meaningful results.
ACKNOWLEDGEMENTS

I would like to thank JR Dennison for being a phenomenal teacher, leader, and mentor. His patience, care, and tenacity for students’ knowledge acquisition both in and out of the classroom is unprecedented and exemplary of an incredible educator. It was only thanks to him and the former work that was established by him and other researchers that I was able to succeed within the short time I spent in his lab.

I also wish to thank Matthew Robertson, Josh Boman and the rest of the Materials Physics Group for their guidance throughout this work and my mentee Heather Allen, for her adeptness for learning and enthusiasm to assist me. Lastly, a thanks to Tammy Rittenour for allowing me to use her facilities to support this work and Fen Ann for guiding me through the many SEM measurements that needed to be done. This research was supported by a Blood Fellowship from the USU Physics Department (TT) and by a Utah NASA Space Grant Graduate Fellowship (MR). I am greatly appreciative of their contributions towards this work.

Tom Keaton
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<td>Description</td>
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<tr>
<td>---------</td>
<td>-------------</td>
</tr>
<tr>
<td>ac</td>
<td>Alternating current</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic force microscopy</td>
</tr>
<tr>
<td>BSEY</td>
<td>Backscattered electron yield</td>
</tr>
<tr>
<td>dc</td>
<td>Direct current</td>
</tr>
<tr>
<td>EDL</td>
<td>Electrostatic dust lofting</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy dispersive x-ray analysis</td>
</tr>
<tr>
<td>FESDL</td>
<td>Forced electrostatic dust lofting (chamber)</td>
</tr>
<tr>
<td>HOPG</td>
<td>Highly orientated pyrolytic graphite</td>
</tr>
<tr>
<td>MPG</td>
<td>Materials Physics Group</td>
</tr>
<tr>
<td>Microns</td>
<td>Micrometers</td>
</tr>
<tr>
<td>PSI</td>
<td>Pounds per square inch</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>SEY</td>
<td>Secondary electron yield</td>
</tr>
<tr>
<td>TEY</td>
<td>Total electron yield</td>
</tr>
<tr>
<td>USU</td>
<td>Utah State University</td>
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</table>
CHAPTER 1

INTRODUCTION

1.1 Background

Yield measurements of granular particulates are a pivotal piece of information necessary for future space exploration as these are what make up the interstellar medium and the regolith of airless, planetary bodies. These micron scale particulates are responsible for expedited degradation of many technologies for future missions to airless, alien bodies, potentially impacting mission critical objectives or success. Dust has plagued missions since the Apollo era. As Harrison Schmitt, geologist and Apollo 17 astronaut, said, "Dust is the No. 1 environmental problem on the Moon."[1] NASA’s goals to return to the Moon for a sustained presence and later to Mars, elevate the importance of dust research, and more specifically Lunar regolith. Indeed, NASA has characterized this as a critical science objective [1,93].

Dust adhesion to technical, biological surfaces like lungs and each other (FIG. 1.1) are enhanced by electrostatic attraction due to charge accumulation on insulating particles and surfaces. By understanding the physics of dust charging properties, we may more reliably model and test dust processes and mitigate risks that dust imposes. This knowledge is also useful for applications outside planetary and Lunar environments, such as the aggregation of the interstellar medium, the evolution of dusty bodies and planetary structures, and dust levitation.
Until now, measurements of granular samples have been incredibly difficult due to the numerous technical, electrostatic charging challenges that plague such attempts. Issues that have been theorized in nature and have been observed experimentally such as electrostatic lofting can easily cause a sample to self-destruct or at best, permanently change its mechanical orientation upon initial beam currents, making further measurements impossible. Since we are now entering a new realm of measurement possibilities, the issue of data integrity also becomes a concern, as there will be limited comparison data to verify the results. Therefore, this report will review a tested and proven method for highly insulative, granular sample preparation and the subsequent electron yield data acquired to verify validity of the methodology. Although the materials are insulative, the methodology may also be extended more trivially to conductive particulates.

FIG. 1.1. ~1.6 µm mean sized, spherical AL₂O₃ particles adhered to the graphite substrate and each other. Image taken post application of a dry nitrogen jet and FESDL chamber processing, showing the forces between particles were strong enough that no lofting would occur during measurements.
1.2 Justification and Limitations

Regardless of material, MPG proposes that whichever granular sample one wishes to study, that the mounting material is some form of graphitic carbon. MPG uses SEM standard, aluminum cored, double-sided, conducting adhesive, carbon tapes. The purpose for the aluminum core is to ensure flatness of the granular sample since pure carbon tapes are prone to surface irregularities at the micro- and nanoscales. Visual differences are obvious via SEM images provided by a vendor (FIG. 1.2). Moreover, carbon tape is used due to the following: MPG has extensive experience in measuring electron yields of numerous, complex materials and uses highly ordered pyrolytic graphite (HOPG) data as a standard for all electron yield measurements. HOPG is a well understood material as it is atomically flat, not easily contaminated, has low yield, and is conductive. Our HOPG data has been compared to the data sets of other research institutions [42] that are also capable of these measurements in order to verify that we are all in agreement. It should be noted that the aluminum cored, graphitic tape we used is not a universal requirement as different

![SEM images](image-url)

**FIG. 1.2.** SEM images depicting the differences between using aluminum cored SEM tape vs non-Al cored tapes [94].
Materials may need to be suspended in liquids other than water that may disintegrate the adhesive layers of the tape. The following sections will outline the water suspension methodology process utilized for this graphitic tape; however, this may easily be extended to many other adhesives and suspension liquids depending on project requirements.

A couple graphite tapes were trialed, one from LADD Research and another from Ted Pella. EY data was taken for both and are compared to the HOPG data (FIG. 1.3). Ultimately, Ted Pella’s brand was chosen as LADD Research was unable to confirm the origins of some material strands embedded within the graphite top surface. Later on, it was discovered via SEM imaging the material was nickel. Although this would’ve sufficed, we wished to choose a foundation with a more homogenous makeup. Moreover, the strands would create a surface topography that would increase the chance of marring accurate data since surface roughness and particle angularity are variables being studied for influence on the yield data.

\[ \langle E_m \rangle \approx 191 \text{ eV} \]
It should be noted that the following methods are only applicable to granular samples up to mean sizes of ~120 µm. At the time of writing, MPG has not yet experimented with larger samples. Moreover, singular layers are only possible for mean sized samples larger than ~30 µm. Any samples ~10 µm or smaller are almost guaranteed to be multilayered with the methodologies we will outline as it is too difficult to ensure otherwise.

1.3 Materials Requirements and Forced Electrostatic Lofting

Table 1.1, lists the required material necessary for a single sample. Multiple samples at various coverages were prepared and the first attempt, regardless of coverage, rarely yielded the desired results, so multiple attempts at each coverage were conducted. It should be noted that for all iterations of sample types we created, (sizes, coverages, layering, etc.) we found it was unnecessary to force electrostatic lofting prior to measurements and that applying dry nitrogen alone was enough to remove loose grains. We attempted forced lofting by constructing an insulating chamber called Forced Electrostatic Dust Lofting (FESDL), as shown in FIG. 1.4. It

<table>
<thead>
<tr>
<th>ID</th>
<th>Item</th>
<th>Qty</th>
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<tbody>
<tr>
<td>0</td>
<td>Sample</td>
<td>1</td>
</tr>
<tr>
<td>1</td>
<td>Gloves</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>Dry Nitrogen</td>
<td>~28 Liters</td>
</tr>
<tr>
<td>3</td>
<td>Graphitic Mount</td>
<td>1 roll</td>
</tr>
<tr>
<td>4</td>
<td>2x Sided Cu Tape</td>
<td>1 roll</td>
</tr>
<tr>
<td>5</td>
<td>AFM Specimen Disc</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>Suspension Fluid</td>
<td>~10 mL</td>
</tr>
<tr>
<td>7</td>
<td>Vial</td>
<td>1</td>
</tr>
<tr>
<td>8</td>
<td>Dropper</td>
<td>1</td>
</tr>
</tbody>
</table>
consisted of a biasing plate to which the sample slug was restrained, and a grounding plate brought near the sample top surface to create a large electric field up to 2 MV/m. This field was a high, yet safe electric field that does not exceed typical dielectric breakdown potentially typically in the 50-100 MV/m range. The plate separation was approximately 0.1 m and the bias was pulsed from 0 to 20 keV at one pulse per 20 sec. Before and after SEM images were taken of the samples that underwent this process and then put through an image analysis program along with visual verification to confirm that there were next to no changes in sample topography.

FIG. 1.3. Diagram of the FESDL chamber used to test if inducing lofting phenomena prior to measurements was necessary.
CHAPTER 2

PROCEDURES AND METHODOLOGY

2.1 Size Characterization and Electron Ranges

Before preparation of the sample, we ensured there were enough particulates for both creating a sample for yield measurements and size characterization. FIG. 2.1 shows the size distribution for a granular alumina sample measured via a Malvern Mastersizer 2000 laser diffractometer at the USU Geology, Luminescence Lab. This required significantly more material than what was needed to make the samples for measurement. For this experiment, size was a parameter we needed to track and even though the sample material was purchased from a manufacturer with a specification, the sizes still needed to be verified via laser diffractometry.

![Statistics Graph (9 measurements)](image)

FIG. 2.1 The distribution width is ~67±23 µm, as shown by the green vertical lines.
Moreover, it is also important to determine if the substrate will have an effect on results due to incident beams penetrating the granular material and interacting with the substrate directly.

In order to verify the incident beams would not permeate the sample surface for full coverages, we verified that the peak of the distribution for particulate size was less than the electron range for the highest, incident energy in the experiment using MPG’s online range tool [97] (FIG. 2.2). If one wishes to be pedantic, standard deviations below the mean value can be used as the limit instead. In this case, no contribution from the aluminum core of the tape was evident. Finally, EDX data was taken on the tape and with fractional coverages (FIG. 2.3) to confirm that only alumina and graphite were the surface materials expected to be measured.
The first step of the preparation process began with us, acquiring a piece of double-sided, graphite tape from a bulk role or a tab role and stamping out the desired diameter. We ensured that the diameter used, typically 10-18 mm, was about 4 mm larger than the incident beam expected to hit the target. This is due to non-uniform coverage near the sample edges being a normality for desired coverages less than ~100%. The non-uniformity stems from when the sample is suspended in the water and deposited onto the tape. Internal, hydrostatic forces within the bubble cause a ring of least concentration to generate near the outer edges of the tape after deposition. An example of

FIG. 2.3. EDX data taken on a fractional coverage (~75%) sample distinguishing the material makeup of the substrate and granular sample.

2.2 Substrate Diameter versus Incident Beam Diameter
this is shown in FIG. 2.4. However, if one is attempting to prepare a ~100% coverage sample, the over sizing may be disregarded.

2.3 Single Layer, Fractional and ~100% Coverages for >10-Micron Particles

When preparing these sample sets, we add in some of the granular sample to a vial filled with deionized water. The density was high enough that after capping the vial and shaking it by hand, we were able to produce a mixture that was non-transparent, but viscous enough to be

FIG. 2.4. Various, relatively uniform coverages of alumina dust mounted onto graphitic carbon tape diameters. Darkened corners on some samples were artifacts of the SEM imaging process. Red circles indicate the approximate area where the incident beam will contact the sample surface. [93]
difficult to pour out. With haste, we took a syringe and extract the mixture before the sample had time to settle in the vial. Then carefully, we positioned the syringe tip vertically and at the center of the tape diameter, to deploy the liquid onto the surface. Due to hydrostatic and surface tension forces the mixture bubbled on the top surface and contained itself within the tape boundaries so long as it was left undisturbed. After extracting the syringe from the liquid bubble, we set the sample aside to evaporate. The granular material suspended in the water settled to the bottom and adhered itself to the tape (FIG. 2.5). The time it took for the sample to fully dry depended on atmospheric humidity and therefore could take anywhere between 4-10 hours. If necessary, one may bakeout the samples after this step, but we opted not to. Once most, if not all, of the suspension fluid evaporated, a uniform, mechanical force was applied directly onto the top, granular surface. This pushed material further into the adhesive. Next, 60 - 100 PSI of dry nitrogen jet was directed onto the sample surface. This ensured that any loose particulates that may have stacked on top of the first layer are removed. A visual change usually occurred, indicating this was an effective step. This should then suffice for any fractional coverage sample we desired. However, for full coverage samples, the following steps were just the first part of the methodology. The reader should skip to Section 2.7 if the desired result is a fractional coverage to complete the final, optional process.

FIG. 2.5. Diagram of deposition process.
2.4 Single Layer, ~100% Coverage for >10-Micron Particles

The prior steps outlined in Section 2.3 for fractional coverages are a prerequisite for 100% coverage. We found that after the dry nitrogen jet was applied, this revealed any areas of the tape that the sample did not adhere to. It was discovered that simply hand dropping more granular material onto the surface was all that was necessary. Afterwards, we angled the sample approximately 45 degrees and tapped the mount by hand, allowing the vibrational forces to move the air deployed material across the sample surface. As the new material moved, individual grains would fall into the cavities that were left after the nitrogen removal process. After most of the new material was vibrated off, we used nitrogen again to remove the rest. We repeated this process at least one more time (for a total of two times) but angled the sample in the opposite direction. However, this step may be repeated any number of times if necessary.

MPG originally attempted to quantify what suspension liquid densities were best for a desired coverage but quickly found that this variable was obfuscated by human error and unnecessary. Factors such as vial mixing, extraction and application time all were variables that were difficult to track. Therefore, unless the process can be automated, this is simply a trial-and-error process for the user. This leaves open an opportunity for others to pursue such quantifications where applicable and desired.

2.5 Multilayer, 100% Coverage for <10-Micron Particles and Nanomaterials

It was found that water deposition alone for sizes below 30 µm worked best (see Section 2.3 for procedure). Even after applying the nitrogen jet to the dried, granular surface, the sample stayed clumped together. Unfortunately, we were not able to yield a method yet for multi-layer,
granular samples for sizes larger than 30 µm as we found the nitrogen jet was satisfactory at removing any grains not directly adhered to the graphite tape.

2.6 ~Single Layer, 100% Coverage for <10-Micron Particles and Nanomaterials

It is exceptionally difficult to acquire a single layer for nanomaterials and any mean sized particles below 10 µm, however, we attempted various methods to try to get the best possible results and found that dropping particles by hand, followed by dry nitrogen exposure sufficed (see Section 2.3 for this procedure).

2.7 Final (Optional) Step for All Sample Types

In order to ensure that electrostatic lofting would not occur with any of the samples we created, we applied a high, dynamic voltage to the sample mount while bringing a grounding plate as close (until dielectric breakdown for air was observed) as possible to the surface of the sample. This produced large electric fields which could potentially force any particulates that were still adhered to the sample surface to loft away via electrostatic forces. However, as stated in Section 2.1, it was found that the affects were negligible as most of the layers above the first were removed by the dry nitrogen jet alone.
CHAPTER 3

SAMPLE COVERAGE VERIFICATION

3.1 >10-Micron Particles

In order to quantify the coverages of the sample material on the tape surfaces, MPG created an analysis tool capable of verifying the SEM images taken. The Matlab program (see appendix A for code) first applies a grayscale color map to the image, then alters the original image so that it becomes more contrasted and sharp prior to analysis (FIG. 3.1). The three variables associated with these pixel alterations are radius, greyscale threshold and amount (sharpening parameter). More information about what each variable alters in Matlab’s built-in function used in the program may be found on Matlab’s site [95]. The purpose of this was so the analysis program yielded more accurate coverage results (FIG. 3.1) when it turned the image into a binary, black and white image. The binary image was created by specifying a greyscale threshold value that placed each pixel into one of two bins, depending on whether it was above or below the threshold. A typical threshold

![Original Image, Post Processed Image, Binary Image](image)

FIG. 3.1. Sub-images taken from the Matlab user interface with the different image types used for analysis. Sample size is ~67±23 µm and the specific sample ID: [0,22.28,50,G1 (CENTER)] based on MPG’s internal sample indexing: [Sample ID, Percent Coverage, Target Coverage, Preparation Method ID (Sub-image location)].
determination example is shown in FIG. 3.2. Any pixels that were below the threshold were assigned a 1 (black value) and any that were above are assigned a 0 (white value). The number of white pixels divided by the total pixels in the image yielded the fractional coverage of the sample surface. The caveat to this method was that in order to ensure results were accurate, the pixel size from the SEM process needed to be smaller than the mean grain size of the sample. For nanomaterials, doing a high resolution, montage image set took too long. Therefore, MPG found an alternative discussed further in Section 3.2.

FIG. 3.2. Example of histogram and binary image threshold selection taken from a 44% coverage surface (see FIG. 2.4).
FIG. 3.3. Grayscale-value histogram for each coverage (shown in FIG. 2.4). For all fractional coverages, there are two distinct peaks representing the material and the tape. For 90%+, there is a single, asymmetric distribution.

3.2 <10-Micron Particles and Nanomaterials

Sizes under 10 µm posed an issue and therefore an alternative method was needed. A high resolution, montage image set whose pixels were smaller than the mean grain size took on the order of 10s of hours, up to a day, depending on instrumentation. Moreover, it was extremely difficult to ensure a single layer for materials under 10 µm.
Therefore, an alternative method was developed where we took the highest resolution SEM montage of the majority of the sample to visually check if the density was relatively uniform (FIG. 3.4). We then took a single, high resolution, enhanced image taken near the center of the sample to ensure the pixels were smaller than the mean grain size and ran it through the image analysis program to verify that subregion’s coverage (FIG. 3.5.). It was then assumed that this coverage was applicable to the rest of the sample surface. The uniformity could be determined visually in the “global image” (FIG. 3.4) along with the projected assumption that regardless of sample size, the density across the surface was radially constant until some radius near the tape edge, where it
drops off. This constant density for the majority of the tape surface was visually apparent for samples with mean grain sizes of \(\sim 67\pm 23 \, \mu m\) and coverage of 50% or lower (FIG. 2.4).

### 3.3 Conclusion

The samples whose data are shown in this chapter were prepared for repeatable, EY measurements. Multiple samples were created using either angular or spherical Al\(_2\)O\(_3\) particulates with narrow distributions for size in attempt to discretize this parameter. Of most relevance however, is figure 2.4 which shows the various, uniform coverages for the \(\sim 67\pm 23 \, \mu m\), Al\(_2\)O\(_3\) material selected for measurements. Each coverage; 22%, 44%, 73%, 91% were prepared as described in Chapter 2. The coverages were uniform in the center region of the samples, where EY measurements are typically made. Coverages were typically lower near the edges, but these regions were not exposed to the beam path and thus had negligible effects on data. We determined the coverages using a custom made, Matlab program which counted the number of illuminated pixels representing a grain area, then divided that by the total number of pixels for the sample surface. Additional samples were prepared for angular Al\(_2\)O\(_3\) particles of sizes: \(\sim 1.6 \, \mu m\) (FIG. 3.4 and 3.5), \(\sim 3.9 \, \mu m\), \(\sim 50 \, \mu m\) and \(\sim 122 \, \mu m\) and various coverages for each. Spherical Al\(_2\)O\(_3\) samples were also created (FIG. 1.1) to understand angularity dependence for yields.
FIG. 3.5. Alternative verification for grain sizes below 10 \( \mu \text{m} \) is to take a single SEM image of the center of the sample-substrate surface (“local image”), then run this image through an image analyzer program. This figure is an example of a local image for a ~1.6 \( \mu \text{m} \) sample. Using the “global image” to visually determine uniformity and the coverage results for the “local image”, assume coverage results apply to the whole sample surface.
We now present an example of this practiced methodology for the purpose of experimental electron yield measurements. In order to ensure that yield results for complete coverage of graphitic carbon tape by alumina dust were accurate, MPG employed a “fractional coverage method”. If there was a clear trend in the data, then this would confirm accuracy. This began by measuring the yield of graphitic carbon tape, without any dust covering it, then continued with measurements of uniform, fractional coverages, which nominally increased by about 25% each. In

![Graph showing secondary electron yields](image)

**FIG. 4.1.** Secondary electron yields of various, fractional coverages of angular, high purity, homogenous alumina particles on graphite tape. [90]
totality, five samples (~0%, ~25%, ~50%, ~75% and ~100% coverage) who’s SEM images are shown in (FIG. 2.4), were measured and curve fits were applied to the data. Figure 4.1 displays the curve fits for the secondary yields of these coverages and figure 4.2 depicts the backscattered electron yield. With the exception of the 73% backscattered data, there is a clear trend between the initial 0% coverage (ie., pure graphitic carbon tape) and the total coverage of the tape, supporting the validity of the alumina dust yield measured. Note, the differences in the backscattered data are

![Graph showing backscattered electron yield](image)

**FIG. 4.2.** Backscattered electron yields of various, fractional coverages of dust on graphite tape.

<table>
<thead>
<tr>
<th>Coverage</th>
<th>$\delta_{max}$</th>
<th>$E_{max}$</th>
<th>m</th>
<th>n</th>
<th>$E_1$</th>
<th>$E_2$</th>
<th>$\eta_{max}$</th>
<th>$E_{max}$</th>
<th>$\eta_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>2.024</td>
<td>160.582</td>
<td>0.426</td>
<td>1.294</td>
<td>21.5</td>
<td>765</td>
<td>0.194</td>
<td>380.953</td>
<td>0.04</td>
</tr>
<tr>
<td>22%</td>
<td>1.801</td>
<td>335</td>
<td>0.58</td>
<td>1.246</td>
<td>58</td>
<td>1090</td>
<td>0.119</td>
<td>416.499</td>
<td>0.0546</td>
</tr>
<tr>
<td>44%</td>
<td>1.66</td>
<td>410</td>
<td>0.43</td>
<td>1.293</td>
<td>83</td>
<td>1357</td>
<td>0.13</td>
<td>440.484</td>
<td>0.0754</td>
</tr>
<tr>
<td>73%</td>
<td>1.7</td>
<td>558</td>
<td>0.477</td>
<td>1.149</td>
<td>115</td>
<td>1881</td>
<td>0.149</td>
<td>600</td>
<td>0.1</td>
</tr>
<tr>
<td>91%</td>
<td>1.92437</td>
<td>610.079</td>
<td>0.094296</td>
<td>1.52848</td>
<td>150</td>
<td>2500</td>
<td>0.18</td>
<td>800</td>
<td>0.0914</td>
</tr>
</tbody>
</table>

**Table 4.1 Key values for SEY and BSEY Fits**
highly exaggerated in the graph in FIG. 4.2 to show the trend. The maximum BSEY and $\eta_{\text{peak}}$ in figure 4.2 vary only from ~0.12 - 0.18, a difference of only ~0.06 BS electrons out per electrons in. These are “fractions of electrons” differences and show how accurate and precise EY measurements from the USU instrumentation are.

Trends are more easily depicted by taking peak energy and yield values as functions of coverage for SEY and BSEY data. Figure 4.3 plots SEY energy at $\delta_{\text{max}} (E_{\text{max}})$, $SEY_{\text{max}} (\delta_{\text{max}})$, BSEY energy at $\eta_{\text{max}}$ and $BSEY_{\text{max}} (\eta_{\text{max}})$ respectively. Values are listed in Table 4.1. Figure 4.3 shows the clear, linear trend of the SEY max energy ($E_{\text{max}}$) values with respect to coverage. The

![SEY Peak Energy and $\eta$ Max Values](image)

FIG. 4.3. Composite plot of all $E_{\text{max}}$ and $\eta_{\text{max}}$ values with respect to coverage. For the $E_{\text{max}}$ values: Polished, bulk sapphire is considered 100% coverage and follows the blue trend line. The same applies to HOPG (green point) when compared to the graphite tape, showing minute differences. For the $\eta_{\text{max}}$ values: The trend is parabolic. [93]
SEY value of $E_{max}$ increases by $\sim 4.98$ eV per % coverage. Additional points for HOPG and polished, bulk alumina are also overplotted in order to further cement this correlation between coverage and energy peak values as they both follow the trend line. The SEY maximum yield ($\delta_{max}$) values follow a parabolic trend (FIG. 4.3) due to the varying magnitudes of constituents of both alumina and carbon linearly combining to create the composite yield peaks.
CHAPTER 5

CONCLUSION

Much is known about interstellar dust and the myriad dangers it poses to space travel from extensive prior investigations of these highly abrasive, insulative and ultra-fine materials (FIG. 5.1) [98-101]. However, a critical knowledge gap exists to address both engineering strategies and basic science issues related to charged dust, namely the EY data that defines the charging properties of these materials in response to space environment charged particle and electromagnetic radiation fluxes. Limited high-quality research conducted since the Apollo missions on EY [17,2,3], ion yields [4,5], photoyields [3,8-12], electron emission spectra, discharge, and luminescence are available for only a few, specific samples. The results of these previous studies are significantly limited by severe charging effects [83,93,102]. Further charging studies of individual Lunar dust grains have indirectly provided results of EY [38-41] and photoyields [30,42,34-37], though the effects of small grain size and shape are not well understood [15,40]; while these studies may be highly relevant to isolated dust grains, many applications

FIG. 5.1. Comparison of SEM images of Lunar dust (left) [103] and a sample prepared at MPG (right).
involve properties of bulk or aggregate dust particles. The preparation methodology conveyed in this report addresses how to create samples that do consider how one may isolate and characterize these non-negligible parameters. Our demonstration has covered more than 50% of the size distribution of Lunar fines as indicated by the green area on Figure 5.2 and is easily applicable for sizes larger than the 120 μm limit we practiced with, up to 3 mm. Moreover, it is also applicable to nanomaterials (Not included in graph’s axis).

Although these studies have proven that such measurements are possible [83,93,102], the most recent researchers conducting such experiments have acknowledged the tremendous challenge of such EY measurements due to the experimental complexities such as a robust sample preparation method [17]. As Duke noted: “The complexity of secondary electron emission is such that no accurate theory [or experiments] exists that can be used in astronomical applications.

**Aggregated Particle Size Data for Various Lunar Samples**

![Graph](image)

- Averaged Data from Various Apollo Samples

FIG. 5.2. Shown here is the aggregated data for size characterizations conducted by various research institutions on samples from Apollo 11, 12 14, 15, 16 and 17 [102]. Red lines indicate the size distribution boundaries, while the green area indicates the range of sizes our sample preparation methodology has been tested for.
Electron emission from electrical insulators, such as most Lunar material, is a difficult topic because of the existence of time-dependent charges with unknown lifetimes and mobility” [17]. Until we have accurate, absolute EY measurements of cosmic dusts and understand the effects of their material properties, modeling and mitigating charge-induced dust processes will be precariously limited. Therefore, now that this sample preparation methodology has been established, tried and proven, this will grant future researchers the opportunity to conduct EY, photoyield and ion yield measurements to fully characterize the electrodynamic, material properties of regolith and the interstellar medium.
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APENDICIES
%User Inputs
FileName = 'matlabtestimage.png'; %Name of image user wants to analyze
GrayScaleThreshold = 90; %Threshold value of the binarization
ParticleSize = 0.3; %Size of particulate (um)
ParticulateSizeError = 'N/A'; %Error range of particle size
%Set SampleDiameter to 0 if image is rectangular
SampleDiameter = 9; %In (mm)
%If image is circular there is no need to set the following values =0 as the
%program will not overwrite the parameters if the SampleDiameter equals a non-zero value
SampleXLength = 9; %Horizontal distance corresponds to # of columns in image matrix (mm)
SampleYLength = 9; %Vertical distance corresponds to # of rows in image matrix (mm)

%User should not be concerned with anything below this line unless they
%wish to add functionality
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

%Conversions
T = GrayScaleThreshold/255;

%Processor
[RGB,cmap,alpha] = imread(FileName); %Here alpha is transparency
I = im2gray(RGB);
%The im2gray function converts RGB images to grayscale
%by eliminating the hue and saturation information while retaining the luminance.

x0 = -1;
x1 = -1;
y0 = -1;
y1 = -1;
[row,col] = size(alpha);
alphaVet = zeros(row,col); %Creates matrix that will be populated by the vetted data cells

%Processing image for clarity prior to application of grayscale value threshold
PostIm = imsharpen(I,'Radius',IRadius,'Amount',IAmount,'Threshold',IThreshold);
BW = imbinarize(PostIm,T); %Converts image to binary where 0=white and 1=black using user
%defined threshold value
BWNNew = uint8(BW);
% For every cell that is not fully transparent, set it equal to 3. Crops photo by pixel
for index1 = 1:row
    for index2 = 1:col
        if alpha(index1,index2) ~= 255
            BWNew(index1,index2) = 3;
        end
    end
end

% The following loops look for the edges of the image using the alpha matrix
% (transparency matrix). It searches for the first non-zero transparency
% cell value and uses those as the edges.
% %%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Pixel size approximation (For X0). Iterates by col.
for index3 = 1:row
    for index4 = 1:col
        if alpha(index3,index4) ~= 0
            x0 = index3;
            break
        end
    end
    if x0 ~= -1
        break
    end
end

% Pixel size approximation (For X1). Iterates by col.
for index5 = 0:row-1
    for index6 = 1:col
        if alpha((row - index5),index6) ~= 0
            x1 = row - index5;
            break
        end
    end
    if x1 ~= -1
        break
    end
end

% Pixel size approximation (For Y0). Iterates by row.
for index7 = 1:col
    for index8 = 1:row
        if alpha(index7,index8) ~= 0
            y0 = index8;
            break
        end
    end
    if y0 ~= -1
        break
    end
end
if alpha(index8, index7) ~= 0
    y0 = index7;
    break
end
end
if y0 ~= -1
    break
end
end

% Pixel size approximation (For Y1). Iterates by row.
for index9 = 0:col-1
    for index10 = 1:row
        if alpha(index10, (col - index9)) ~= 0
            y1 = col - index9;
            break
        end
    end
    if y1 ~= -1
        break
    end
end

% Calculations
NoOnesIndex = BWNew == 1;
NoZerosIndex = BWNew == 0;
NoOnes = sum(NoOnesIndex(:)); % Carbon tape exposure (black pixels)
NoZeros = sum(NoZerosIndex(:)); % Particulate coverage (white pixels)
TotalSum = NoOnes + NoZeros;
CoveragePerc = round((NoOnes/TotalSum)*100, 2);

% Pixel Size Calc
if SampleDiameter == 0
    SampleXSetSize = SampleXLength;
    SampleYSetSize = SampleYLength;
else
    SampleXSetSize = SampleDiameter;
    SampleYSetSize = SampleDiameter;
end

XPixNum = x1 - x0;
YPixNum = y1 - y0;
XPixLength = (SampleXSetSize/XPixNum)*1E+3;
YPixLength = (SampleYSetSize/YPixNum)*1E+3;

% Plots
figure
subplot(2,4,2)
imshow(RGB)
title('Original Image')

subplot(2,4,3)
imshow(PostIm)
title('Post Processed Image')

subplot(2,4,[4;8])
imhist(I)
title('Grayscale Histogram for Post Processed Image')

subplot(2,4,6)
imshow(alpha)
title({'Alpha Matrix Visual for Inclusive Pixel Verification',...'
  (White area indicates pixels that are counted)'})

subplot(2,4,7)
imshow(BW)
title('Binary Image Used in Percent Coverage Computation')

% Text on Plots
str1 = {'\textbf{Percent Coverage}: ', num2str(CoveragePerc), '%'};
annotation('textbox', [0.025, 0.8, 0.1, 0.1], 'FitBoxToText', 1, 'String', str1);

str0 = {'\textbf{Sample Characteristics}',...'
  \textbf{Particulate Size}: ', num2str(ParticleSize), ' micron',...'
  \textbf{Sample Diameter}: ', num2str(SampleDiameter), ' mm',...'
  \textbf{X-Pixel Length (Horizontal)}: ', num2str(XPixLength), ' microns/pixel',...'
  \textbf{Y-Pixel Length (Vertical)}: ', num2str(YPixLength), ' microns/pixel'};
annotation('textbox', [0.025, 0.7, 0.1, 0.1], 'FitBoxToText', 1, 'String', str0);

str2 = {'\textbf{Binary Image Statistics}',...'
  \textbf{Gray scale threshold value for binary processing}: ',...'
  \textbf{White Pixels}: ', num2str(NoOnes), ' Black Pixels: ',...'
  \textbf{Total Pixels Counted}: ', num2str(TotalSum)};
annotation('textbox', [0.025, 0.3, 0.1, 0.1], 'EdgeColor', 'black', 'String', str2);

str3 = {'\textbf{Image Processing Parameters}',...'
  \textbf{Standard deviation of the Gaussian lowpass filter}: ',...'
  \textbf{Strength of the sharpening effect}: ',...'
  \textbf{Minimum contrast required for edge pixel}: '};
annotation('textbox', [0.025, 0.5, 0.1, 0.1], 'EdgeColor', 'black', 'String', str3);